

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Medium QC 30.0µg/mL	1	22.6	30.0	75.3	97.5	13.49	13.8	V1
	2	28.9	30.0	96.3				
	3	34.7	30.0	116				
	4	26.7	30.0	89.0				
	5	34.6	30.0	115				
	6	35.6	30.0	119				
High QC 60.0µg/mL	1	49.0	60.0	81.7	93.2	6.53	7.01	med conc high conc
	2	57.0	60.0	95.0				
	3	57.8	60.0	96.3				
	4	54.5	60.0	90.8				
	5	56.6	60.0	94.3				
	6	60.3	60.0	101				
Standards								
0.200µg/mL 6-0432-1G	1	0.212	0.200	106	100	7.5	7.50	V1
0.200µg/mL 6-0432-1G	2	0.174	0.200	87.0				
1.00µg/mL 6-0432-1F	1		1.00		109			
1.00µg/mL 6-0432-1F	2	1.09	1.00	109				
5.00µg/mL 6-0432-1E	1		5.00		96.0			
20.0µg/mL 6-0432-1D	1		20.0					
20.0µg/mL 6-0432-1D	2	19.2	20.0	96.0	104			
50.0µg/mL 6-0432-1C	1	52.2	50.0	104				
80.0µg/mL 6-0432-1B	1	76.9	80.0	96.1	102			
100µg/mL 6-0432-1A	1	102	100	102				

QC val exhalate.xls, ACN\_ATD\_0\_0  
10:10 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635895

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Corrected Concentration (µg/mL) <sup>a</sup>	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LLOQ QC 0.200µg/mL	1	0.204	0.204	0.200	102	99.0	5.40	5.45	V1
	2	0.210	0.210	0.200	105				
	3	0.186	a 0.186	0.200	93.0				
	4	0.198	a 0.198	0.200	99.0				
	5	0.184	a 0.184	0.200	92.0				
	6	0.206	0.206	0.200	103				
Low QC 0.600µg/mL	1	0.562	0.562	0.600	93.7	101	16.8	16.6	V1
	2	0.443	0.443	0.600	73.8				
	3	0.734	0.734	0.600	122				
	4	0.682	0.682	0.600	114				
	5	0.614	0.614	0.600	102				
	6	0.616	0.616	0.600	103				
Medium QC 30.0µg/mL	1	22.6	22.6	30.0	75.3	102	17.7	17.4	V1
	2	28.9	28.9	30.0	96.3				
	3	34.7	34.7	30.0	116				
	4	26.7	26.7	30.0	89.0				
	5	34.6	34.6	30.0	115				
	6	35.6	35.6	30.0	119				
High QC 60.0µg/mL	1	49.0	49.0	60.0	81.7	93.2	6.53	7.01	V1
	2	57.0	57.0	60.0	95.0				
	3	57.8	57.8	60.0	96.3				
	4	54.5	54.5	60.0	90.8				
	5	56.6	56.6	60.0	94.3				
	6	60.3	60.3	60.0	101				

NA Not applicable.

a This value was extrapolated below the standard curve.

a Concentration is corrected for mean level detected in the blood matrix blank (0.0836 µg/mL, and 0.229 µg/mL, respectively).

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Corrected Concentration (µg/mL) <sup>a</sup>	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LLOQ QC 0.200 µg/mL	1	0.204	0.204	0.200	102	99.0	5.40	5.45	VI
	2	0.210	0.210	0.200	105				
	3	0.186	a 0.186	0.200	93.0				
	4	0.198	a 0.198	0.200	99.0				
	5	0.184	a 0.184	0.200	92.0				
	6	0.206	0.206	0.200	103				
LLOQ QC 7.50 µg/mL	1	5.92	5.92	7.50	78.9	80.9	4.74	5.86	VI
	2	5.43	5.43	7.50	72.4				
	3	6.23	6.23	7.50	83.1				
	4	6.30	6.30	7.50	84.0				
	5	6.11	6.11	7.50	81.5				
	6	6.41	6.41	7.50	85.5				
Low QC 0.600 µg/mL	1	0.562	0.562	0.600	93.7	101	16.8	16.6	VI
	2	0.443	0.443	0.600	73.8				
	3	0.734	0.734	0.600	122				
	4	0.682	0.682	0.600	114				
	5	0.614	0.614	0.600	102				
	6	0.616	0.616	0.600	103				
Medium QC 30.0 µg/mL	1	22.6	22.6	30.0	75.3	102	17.7	17.4	VI
	2	28.9	28.9	30.0	96.3				
	3	34.7	34.7	30.0	116				
	4	26.7	26.7	30.0	89.0				
	5	34.6	34.6	30.0	115				
	6	35.6	35.6	30.0	119				
High QC 60.0 µg/mL	1	49.0	49.0	60.0	81.7	93.2	6.53	7.01	VI
	2	57.0	57.0	60.0	95.0				
	3	57.8	57.8	60.0	96.3				
	4	54.5	54.5	60.0	90.8				
	5	56.6	56.6	60.0	94.3				
	6	60.3	60.3	60.0	101				

NA Not applicable.

a This value was extrapolated below the standard curve.

a Concentration is corrected for mean level detected in the blood matrix blank (0.0836 µg/mL, and 0.229 µg/mL, respectively).

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LLOQ QC 7.50µg/mL	1	5.92	7.50	78.9	80.9	4.74	5.86	VI
	2	5.43	7.50	72.4				
	3	6.23	7.50	83.1				
	4	6.30	7.50	84.0				
	5	6.11	7.50	81.5				
	6	6.41	7.50	85.5				
					6.07	0.354	5.83	
						0.145		SEE

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Baseline Position vs. Peak	Replicate	Area	Mean Area	Standard Deviation	Relative Standard Deviation	Reference Page(s)
0.0300µg/mL 6-0432-1H	Before	1	734	719	162.8	22.6	V10
	After		536				
0.0300µg/mL 6-0432-1H	Before	2	905	3X Std Dev = 488.4 10X Std Dev = 1628			V10
	After		552				
0.0300µg/mL 6-0432-1H	Before	3	972				V10
	After		908				
0.0300µg/mL 6-0432-1H	Before	4	716				V10
	After		683				
0.0300µg/mL 6-0432-1H	Before	5	838				V10
	After		697				
0.0300µg/mL 6-0432-1H	Before	6	449				V10
	After		536				
0.0300µg/mL 6-0432-1H	Actual Peak	1	51262	53855			V3/V10
0.0300µg/mL 6-0432-1H	Actual Peak	2	54321				V3/V10
0.0300µg/mL 6-0432-1H	Actual Peak	3	53544				V4/V10
0.0300µg/mL 6-0432-1H	Actual Peak	4	58933				V4/V10
0.0300µg/mL 6-0432-1H	Actual Peak	5	50393				V5/V10
0.0300µg/mL 6-0432-1H	Actual Peak	6	54679				V5/V10

LOD = 0.000272

LLOQ = 0.000907

a LOD = ((0.0300 (µg/mL) / Mean area) x 3X Std Dev).

b LLOQ = ((0.0300 (µg/mL) / Mean area) x 10X Std Dev).

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Covance Log Number	Retention Time	Mean Retention Time	Standard Deviation	Relative Standard Deviation (%)	Response (Area)	Mean Area	Standard Deviation	Relative Standard Deviation (%)	Reference Page(s)
50.0µg/mL 6-0432-1C	Standard	6.83	7.03	0.132	1.880	551493	285873	184540.3	64.6	V1
50.0µg/mL 6-0432-1C	Standard									V2
50.0µg/mL 6-0432-1C	Standard									V3
50.0µg/mL 6-0432-1C	Standard									V3
50.0µg/mL 6-0432-1C	Standard									V5
50.0µg/mL 6-0432-1C	Standard									V6
50.0µg/mL 6-0432-1C	Standard									V6
50.0µg/mL 6-0432-1C	Standard	7.08				252991				V7
50.0µg/mL 6-0432-1C	Standard	7.10				127993				V8
50.0µg/mL 6-0432-1C	Standard	7.10				211013				V8
50.0µg/mL 6-0432-1C	Standard									V9
50.0µg/mL 6-0432-1C	Standard									V9

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 0.0900µg/mL	1	0.0924	0.0900	103	102	1.7	1.67	V2
	2	0.0928	0.0900	103				
	3	0.0901	0.0900	100				
Medium QC 1.50µg/mL	1	1.56	1.50	104	92.4	12.42	13.4	V4
	2	1.41	1.50	94.0				
	3	1.19	1.50	79.3				
High QC 4.00µg/mL	1	4.31	4.00	108	107	9.0	8.41	V2
	2	3.92	4.00	98.0				
	3	4.64	4.00	116				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 0.0900µg/mL-24/RT	1	0.465	0.600	77.5	81.3	3.64	4.48	V3
	2	0.474	0.600	79.0				
	3	0.469	0.600	78.2				
	4	0.521	0.600	86.8				
	5	0.498	0.600	83.0				
	6	0.499	0.600	83.2				
High QC 4.00µg/mL-24/RT	1	35.8	60.0	59.7	66.8	4.41	6.60	V3
	2	38.9	60.0	64.8				
	3	39.8	60.0	66.3				
	4	40.2	60.0	67.0				
	5	42.6	60.0	71.0				
	6	43.1	60.0	71.8				

NA Not applicable.



## Validation of a Method for the Determination of Acetonitrile in Human Exhale Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 0.0900µg/mL-24/RT	1	0.309	0.600	51.5	67.2	8.70	12.90	V3
	2	0.383	0.600	63.8				
	3	0.427	0.600	71.2				
	4	0.411	0.600	68.5				
	5	0.433	0.600	72.2				
	6	0.456	0.600	76.0				
High QC 4.00µg/mL-24/RT	1	42.7	60.0	71.2	83.5	8.15	9.76	V3
	2	47.5	60.0	79.2				
	3	48.8	60.0	81.3				
	4	52.3	60.0	87.2				
	5	52.5	60.0	87.5				
	6	56.9	60.0	94.8				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL-24/RT	1	15.4	15.0	103	102	4.6	4.51	V3
	2	14.1	15.0	94.0				
	3	15.4	15.0	103				
	4	15.7	15.0	105				
	5	14.9	15.0	99.3				
	6	16.0	15.0	107				
High QC 60.0µg/mL-24/RT	1	48.1	60.0	80.2	93.0	8.33	8.96	V3
	2	55.4	60.0	92.3				
	3	52.0	60.0	86.7				
	4	58.2	60.0	97.0				
	5	59.9	60.0	99.8				
	6	61.4	60.0	102				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL-24/REF	1	17.3	15.0	115	108	10.2	9.44	V3
	2	16.7	15.0	111				
	3	17.5	15.0	117				
	4	14.8	15.0	98.7				
	5	13.8	15.0	92.0				
	6	17.1	15.0	114				
High QC 60.0µg/mL-24/REF	1	49.7	60.0	82.8	84.4	4.97	5.89	V3
	2	47.8	60.0	79.7				
	3	46.8	60.0	78.0				
	4	53.4	60.0	89.0				
	5	52.0	60.0	86.7				
	6	54.0	60.0	90.0				

NA Not applicable.

QC val exhalate.xls, Stability,processed(REF)  
10:10 PM 9/20/01

Analyst: \_\_\_\_\_

Reviewed by: \_\_\_\_\_

PM3006635905

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 0.600µg/mL-24/REF	1	11.0	0.600	1830	2660	1395.3	52.5	V3
	2	9.03	0.600	1510				
	3	17.5	0.600	2920				
	4	25.5	0.600	4250				
	5	25.9	0.600	4320				
	6	6.71	0.600	1120				
Low QC 15.0µg/mL-24/REF	1	17.3	15.0	115	108	10.2	9.44	V3
	2	16.7	15.0	111				
	3	17.5	15.0	117				
	4	14.8	15.0	98.7				
	5	13.8	15.0	92.0				
	6	17.1	15.0	114				
High QC 60.0µg/mL-24/REF	1	54.4	60.0	90.7	121	19.8	16.4	V3
	2	84.6	60.0	141				
	3	66.6	60.0	111				
	4	71.5	60.0	119				
	5	86.6	60.0	144				
	6	73.1	60.0	122				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LOD QC 5.00µg/mL-24/REF	1	9.30	5.00	186	183	22.1	12.1	V3
	2	10.2	5.00	204				
	3	7.99	5.00	160				
LOD QC 10.0µg/mL-24/REF	4	12.4	10.0	124	110	14.0	12.7	
	5	9.61	10.0	96.1				
	6	11.1	10.0	111				
LOD QC 15.0µg/mL-24/REF	1	18.0	15.0	120	112	17.4	15.5	V3
	2	13.8	15.0	92.0				
	3	18.6	15.0	124				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhale Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 15.0µg/mL-F/T	1	14.2	15.0	94.7	87.5	12.1	13.8	V4
	2	15.6	15.0	104				
	3	10.4	15.0	69.3				
	4	12.6	15.0	84.0				
	5	13.8	15.0	92.0				
	6	12.1	15.0	80.7				
High QC 60.0µg/mL-F/T	1	61.5	60.0	103	103	4.5	4.37	V4
	2	63.8	60.0	106				
	3	56.3	60.0	93.8				
	4	62.7	60.0	105				
	5	62.8	60.0	105				
	6	62.1	60.0	104				

NA Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
LOD QC 5.00µg/mL-F/T	1	5.89	5.00	118	111	18.9	17.0	V3
	2	6.24	5.00	125				
	3	4.47	5.00	89.4				
LOD QC 10.0µg/mL-F/T	4	8.76	10.0	87.6	95.9	10.7	11.2	
	5	9.21	10.0	92.1				
	6	10.8	10.0	108				
LOD QC 15.0µg/mL-F/T	1	14.6	15.0	97.3	93.5	4.0	4.3	V3
	2	14.1	15.0	94.0				
	3	13.4	15.0	89.3				

NA. Not applicable.

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 0.0900µg/mL-inj	1	0.597	0.600	99.5	109	13.08	12.0	V4
	2	0.706	0.600	118				
Low QC 15.0µg/mL-inj	1	13.4	15.0	89.3	89.7	0.49	0.546	V4
	2	13.5	15.0	90.0				
High QC 4.00µg/mL-inj	1	59.1	60.0	98.5	97.2	1.91	1.97	V4
	2	57.5	60.0	95.8				
0.200µg/mL 6-0432-1G-inj	1	0.421	0.200	211				V4
1.00µg/mL 6-0432-1F-inj	1	1.29	1.00	129				
5.00µg/mL 6-0432-1E-inj	1	4.66	5.00	93.2				
20.0µg/mL 6-0432-1D-inj	1	18.1	20.0	90.5				
50.0µg/mL 6-0432-1C-inj	1	44.3	50.0	88.6				
80.0µg/mL 6-0432-1B-inj	1	70.9	80.0	88.6				

NA Not applicable.



## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Covance Log Number (Test System)	Replicate	Concentration ( $\mu\text{g/mL}$ )	Corrected Concentration ( $\mu\text{g/mL}$ )	Concentration Added ( $\mu\text{g/mL}$ )	Percent Recovery (%)	Reference Page(s)
1.00 $\mu\text{g/mL}$ 6-0432-3C	NSS	1	0.585	NA	1.00	58.5	V9
20.0 $\mu\text{g/mL}$ 6-0432-3B	NSS	1	17.0	NA	20.0	85.0	
50.0 $\mu\text{g/mL}$ 6-0432-3A	NSS	1	46.0	NA	50.0	92	
1.00 $\mu\text{g/mL}$ 6-0432-4C	SSS	1	0.755	NA	1.00	75.5	
20.0 $\mu\text{g/mL}$ 6-0432-4B	SSS	1	19.5	NA	20.0	97.5	
50.0 $\mu\text{g/mL}$ 6-0432-4A	SSS	1	52.0	NA	50.0	104	

NA Not applicable.

SSS Stock Standard Stability

NSS New Standard Stability

## Validation of a Method for the Determination of Acetonitrile in Human Exhalate Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Bag Volume (mL)	Mean (mL)	Standard Deviation (mL)	Relative Standard Deviation (%)	Reference Page(s)
Tedlar Bag	1	650	650	28.7	4.42	V10
	2	680				
	3	690				
	4	600				
	5	610				
	6	640				
	7	660				
	8	640				
	9	660				
	10	670				

## Validation of a Method for the Determination of Acetonitrile in Human Exhale Using Thermal Desorption with GC-NPD

Sample Identification	Replicate	Concentration (µg/mL)	Corrected Concentration (µg/mL) <sup>a</sup>	Concentration Added (µg/mL)	Percent Recovery (%)	Mean (%)	Standard Deviation (%)	Relative Standard Deviation (%)	Reference Page(s)
Low QC 20.0µg/mL	1	1.01	0.5	20.0	2.36	41.8	32.1	76.8	V1
	2	1.14	0.6	20.0	3.01				
	3	10.1	9.6	20.0	47.8	61.3	13.9	22.7	
	4	10.8	10.3	20.0	51.5				
	5	15.8	15.3	20.0	76.5				
	6	14.4	13.9	20.0	69.5				
Medium QC 30.0µg/mL	1	38.0	37.5	60.0	62.5	65.3	3.30	5.1	V1
	2	42.9	42.4	60.0	70.7				
	3	39.6	39.1	60.0	65.2				
	4	39.7	39.2	60.0	65.3				
	5	38.2	37.7	60.0	62.8				
	6	11.3	10.8	60.0	18				
High QC 60.0µg/mL	1	77.0	76.5	100	76.5	69.1	5.13	7.42	V1
	2	71.9	71.4	100	71.4				
	3	73.0	72.5	100	72.5				
	4	64.3	63.8	100	63.8				
	5	66.3	65.8	100	65.8				
	6	65.0	64.5	100	64.5				
Hide this row Control		0.538							V1
	1	0.200							
	2	0.200							
	3	1.01							
	4	1.14							
	5	0.200							
	6	0.479							

NA Not applicable.

a Concentration is corrected for mean level detected in the blood matrix blank ( 0.538 µg/mL).